

Lightweight carbon-bonded carbon fiber composites with quasi-layered and network structure



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ABSTRACT

Lightweight carbon-bonded carbon fiber (CBCF) composites were fabricated with chopped carbon fibers and dilute phenolic resin solution by pressure filtration, followed by carbonization at 1000 °C in argon. The as-prepared CBCF composites had a homogenous fiber network distribution in xy direction and quasi-layered structure in z direction. The pyrolytic carbon derived from phenolic resin was mainly accumulated at the intersections and surfaces of chopped carbon fibers. The composites possessed compressive strengths ranged from 0.93–6.63 MPa in xy direction to 0.30–2.01 MPa in z direction with a density of 0.162–0.381 g cm⁻³. The thermal conductivity increased from 0.314–0.505 to 0.139–0.368 Wm⁻¹ K⁻¹ in xy and z directions, respectively. The experimental results indicate that the CBCF composites prepared by this technique can significantly contribute to improve the thermal insulation and mechanical properties at high temperature.

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1. Introduction

Since developed in the 1960s at Oak Ridge National Laboratory, carbon-bonded carbon fiber (CBCF) composites have been widely used as thermal insulating material and carbonaceous reinforcement of composites, due to its high porosity, low thermal conductivity, and dimensional stability [1–3]. CBCF composites often consist of a chopped carbon fiber network bonded together at the intersections of fibers by discrete regions of carbon matrix, in which the vast majority of the volume consists of interconnected pores (70–90%) [4,5]. Most CBCF composites were prepared using chopped carbon fibers and phenolic resin prepared by vacuum filtration [6–10] or pressure filtration [11]. However, many barriers remain owing to the inhomogeneous distribution of carbon fibers and phenolic resin powder in the green body of CBCF composites, which eventually leads to poor mechanical and thermal performance. The mechanical property of CBCF composites (with an average compressive strength of around 0.8 MPa) [6] could be enhanced by increasing the density of composites as the addition of recycled fiber, however it degraded thermal insulation performance to an unacceptable level [6,12,13]. Another effective approach was depositing pyrolytic carbon coating on fiber surface by chemical vapor deposition process, while it will consume hundreds of hours to achieve the targeted density [14].

Herein, we developed a feasible and novel procedure to prepare CBCF composites. In this approach, the chopped carbon fibers were

directly dispersed in dilute phenolic resin solution instead of phenolic resin powder/water mixture, which can give rise to the microscopic homogeneity and no appearance of agglomeration of as-prepared product. The effects of phenolic resin solution and bulk density of as-prepared CBCF on the microstructure, mechanical and thermophysical properties of the final composites were investigated.

2. Materials and methods

Rayon-based carbon fibers (amorphous structure), phenolic resin solution (60% w/w ethanol solution) and ethanol (analytical grade) were used to fabricate CBCF composites. Fig. 1 shows the schematic illustration of the prepared process.

Typically, the phenolic solution was first diluted by ethanol to the predetermined concentration, and then the continuous fibers with a density of 1.6 g cm⁻³ were cut into segments of 1.5 mm-length and added into the solution, and mechanically stirred for 60 min to make a homogeneous distribution. The fiber-resin slurry was transferred into the metal mold, and the excessive phenolic solution was extruded out by applying pressure on the perforated squeeze head. To keep the shape and size of composites unchanged during follow-up processes, the mold was fixed with screws, and then placed into an air-circulated oven and dried at 80 °C for 24 h to remove the ethanol solvent, followed by curing using the schedule of 120 °C/1 h, 150 °C/1 h and 180 °C/3 h. Finally the cured sample was demolded and carbonized at 1000 °C in argon atmosphere for 1 h.

In order to investigate the effect of the density of CBCF on thermo-mechanical property, four kinds of samples with different densities of

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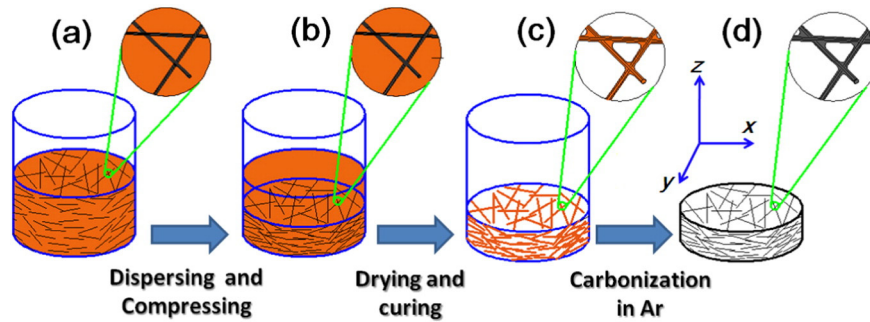


Fig. 1. Schematic illustration of the prepared process for CBCF composites. (a) Chopped carbon fibers were dispersed in dilute phenolic resin solution; (b) the mixture solution was extruded out by pressure infiltration; (c) phenolic-bonded carbon fiber composites after drying and curing; (d) CBCF composites carbonized at 1000 °C for 1 h in Ar.

0.162, 0.251, 0.321 and 0.381 g cm⁻³ were prepared according the experimental condition listed in Table 1.

The microstructure was examined using scanning electron microscopy (FEI HELIOS NanoLab 600i). Compression testing was carried out on an Instron 5569 universal test machine using at least five specimens with dimensions of 10 × 10 × 12 mm³ for each sample, and the loading rate was 0.5 mm min⁻¹. Thermal conductivity was tested on NETZSCH LAF427 using disc-shaped specimen with a diameter of 12.7 mm and a thickness of 2.0 mm in argon between 25 °C and 1275 °C.

3. Results and discussion

Fig. 2 shows the microstructure of CBCF composites with different bulk densities. The as-prepared composites have a homogeneous morphology without cracks and lamination and the majority of the volume consists of interconnected and open pores. The composites possess 2D planar random microstructure in xy direction (shown in Fig. 2(a)–(d)), and the carbon fibers are bonded to each other by the PyC owing to carbonization yield of phenolic resin. Noticeably, a quasi-layered structure in z direction is clearly found from Fig. 2(e)–(h), and the PyC is mainly accumulated at the junctions of fibers to make a bonded bridge adjacent to fiber surfaces (shown in Fig. 2(i)). Furthermore, the PyC formed a thin film coated on the surface of fibers (shown in Fig. 2(j)). Because carbon fibers were totally immersed dilute phenolic resin, the surface of fibers was occupied and coated by phenolic resin sufficiently. Thus, a thin phenolic film was formed after thermal curing and subsequently a carbonaceous PyC film was obtained after thermal pyrolysis.

Fig. 3 shows the representative compressive stress–strain curves of CBCF composites. Unlike typical brittle fracture mode of fibrous ceramics, the fracture mode is a representative ductile one. Two different deformation responses can be observed in these two directions. The compressive stresses in the initial region increase linearly up to the maximum elastic stress (marked by point A), which is defined as compressive strength. The compressive strength increases from 0.93 to 6.63 MPa in xy direction and 0.3 to 2.01 MPa in z direction with densities of 0.162, 0.251, 0.321 and 0.381 g cm⁻³, respectively. The elastic deformation is believed to form through elastic bending and rotation

of reinforcing fibers. It is easy to understand that most chopped carbon fibers in xy direction are distributed with a wide variety of angles to the compressive loading and thus it is helpful to increase the compressive strength through fiber buckling. The successive loading might produce permanent damage in xy direction (Fig. 3(a)), owing to the damage of large quantities of intersections bonded by PyC among fibers. However, the stresses in z direction fluctuate smoothly with strain exceeding 30% (Fig. 3(b)). Since a small amount of junction spots bonded by PyC, loading force can be transferred and rearranged through fibers and therefore the compaction and densification of fibers can bear large deformation in z direction.

Fig. 3(c) shows the thermal conductivity of CBCF composites with different densities and temperature. At room temperature, the thermal conductivity increases from 0.139–0.368 Wm⁻¹ K⁻¹ (for z direction) to 0.314–0.505 Wm⁻¹ K⁻¹ (for xy direction), respectively, as the density varied from 0.162 to 0.381 g cm⁻³. The chopped carbon fibers are overlapped each other and formed “fiber–fiber” pattern, which helped reduce heat-bridge effect compared to long-fiber reinforced composites. Noticeably, in z direction, carbon fibers appear to be aligned and form a quasi-layered structure perpendicular to the direction of heat flow, which can enhance the thermal resistance in this direction due to the existence of limited junction points in the interlayer (shown in Fig. 2(i)). However, in xy direction, the fibrous architecture of CBCF consisted of continuous chopped carbon fiber network, which can enhance the long-path thermal transfer and result in higher thermal conductivity in the inner layer.

Since the deduction of gas-phase heat conduction and thermal radiation is negligible at room temperature. However, at high temperature (25–1275 °C), the thermal conductivity increases from 0.204 to 0.894 Wm⁻¹ K⁻¹ (for z direction) and 0.314 to 1.888 Wm⁻¹ K⁻¹ (for xy direction) (Fig. 3(d)), which is mainly attributed to the thermal radiation and gas conductivity contribution to the overall thermal conductivity at higher temperature.

4. Conclusions

Lightweight, quasi-layered and network CBCF composites without agglomeration and layering were prepared with chopped carbon fibers

Table 1
Experimental conditions of CBCF composite preparation.

Bulk density of CBCF (g cm ⁻³)	Porosity (%)	Diameter of sample (mm)	Thickness of sample (mm)	Weight of carbon fibers (g)	Concentration of phenolic solution (g ml ⁻¹)
0.162	90.1%	70	50	19.233	0.107
0.251	84.7%	70	50	28.849	0.166
0.321	80.4%	70	50	38.465	0.229
0.381	76.7%	70	50	48.081	0.296

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